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OFFICE OF NAVAL RESEARCH

Contract No. N5-ori-07881

NR-039-007

- I. Deformation Studies of Metals at Elevated Temperatures
- II. The Iron-Chromium-Nickel Ternary System
- III. Effect of Structure and Composition on the Strength Properties of Stainless Steel

PERIODIC STATUS REPORT NO. 5

August 1953 - November 1953

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
DEPARTMENT OF METALLURGY
CAMBRIDGE, MASSACHUSETTS

Submitted by:
H. G. Chang
F. C. Monahan
Peter Price
N. J. Grant

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I. Deformation Studies of Metals at Elevated Temperatures

At the beginning of this past quarter it was expected that the new optical furnace (maximum temperature 1600 - 1800 degrees F) would be ready for use. With the tests of the special deoxidized 80 nickel - 20 chromium alloy supplied by the International Nickel Company, it was hoped that the nature of intercrystalline fracture could be determined and that the previously formulated theory of intercrystalline fracture could be put into a more definite form. However, the problem of leakage around the furnace window has resisted solution up to this time. Sealing the window with wax or glyptol has failed because of the high temperature. At the present, sealing with a high temperature rubber gasket is being tried.

The program of the nichrome alloys is delayed because the new optical furnace is not yet in working condition. Instead, to keep other tests under way, a high purity aluminum specimen has been tested at 900 degrees F in the old optical creep furnace in an attempt to determine the effect of grain boundary shape and orientation on the direction of grain boundary migration. There were twelve grains in the specimen, whose orientations were determined by X-ray techniques before the test. The range of orientations covered practically the whole area of a standard triangle on a stereographic projection. After a careful study of the direction of grain boundary migration, the inclination of the grain boundaries with respect to the direction of the applied stress and to the direction of the grain boundary curvature is still the determining factor, which governs the direction of boundary migration during creep, as has been discussed in a previous publication⁽¹⁾. The effect of orientation differences across various grain boundaries appears to be insignificant. In order to ascertain whether this is true for a wide range of orientation differences and geometries, more specimens will be tested.

(1) H. C. Chang and N. J. Grant: Grain Boundary Sliding and Migration and Intercrystalline Failure Under Creep Conditions. Trans. ADME (1953), 197, p. 305, Journal of Metals, (February, 1953).

II. The Iron-Chromium-Nickel Ternary System

The arc melting furnace (cold crucible), heat treating furnaces, hot X-ray camera and auxiliary equipment for this program are now ready for the program. In the meantime, some research is being completed on 18-8 types of steels in which the structure composition and high temperature properties are being related. A description of this work follows:

Research has been carried on for the past year to determine the effects of composition and structure on the properties of 18-8 type stainless steels with the objective of obtaining optimum structures in this type of alloy for high temperature applications. The work of Hum⁽²⁾ indicated that certain acicular structures in 18-8 type steels have creep and rupture properties significantly better than those of single phase austenitic alloys. Hum's structures were obtained, however, by vacuum melting the alloys, which yielded low values of carbon and nitrogen in the steel. In an attempt to learn more regarding these relatively stable acicular structures and feasible methods, a wide range of alloy compositions were made for the purpose. Steels of the following compositions were melted in an induction furnace and cast into thirty-pound ingots:

<u>Steel Number</u>	<u>% Chromium</u>	<u>% Nickel</u>	<u>% Carbon</u>	<u>% Nitrogen</u>	<u>% (Carbon + Nitrogen)</u>
12	18.24	10.97	0.104	0.035	0.139
13	18.28	11.18	0.067	0.035	0.102
11	18.68	10.92	0.032	0.036	0.068
10	18.49	6.09	0.116	0.035	0.151
29	18.66	5.93	0.070	0.035	0.105
1	18.50	6.15	0.032	0.034	0.066

The ingots were forged to 1/2 inch diameter round rods. Nine inch lengths of the rods were solution treated at 2000 degrees F for 1/2 hour and water quenched.

(2) Hum, J.K.Y. and Grant, N. J.: Austenitic Stability and Creep Rupture Properties of 18-8 Stainless Steels. Trans. ASM, Vol. 45, 1953.

Standard test specimens for stress-rupture tests were machined from the rods.

Steels number 12, 13, and 11 are all single phase austenitic steels.

Alloy 10 is austenitic with some martensite. Alloy 29 contains martensite, austenite, and small amounts of ferrite.

Alloy 1 contains martensite, austenite, and large amounts of ferrite.

From the results of stress-rupture tests it was determined that these structures produce large differences in rupture strengths.

The M_s temperatures of the three martensitic steels were determined using the electrical resistivity technique. Experimental values of the M_s points are given in the following table:

<u>Steel</u>	<u>M_s Experimental</u>	<u>M_s Calculated</u>
1	+136° F	+90° F
29	+41° F	+79° F
10	-156° F	-109° F

M_s calculated values were based on the formula determined by Hull and Eichelman (3)

$$M_s^{\circ}F = 75(14.6 - \%Cr) + 110(8.9 - \%Ni) + 50(0.47 - \%Si) + 3000(0.068 - \%C + \%N) + 60(1.13 - \%Mn) \pm 60^{\circ}F$$

On the basis of the M_s for steel 10, the martensite in the structure must be due to quenching strains.

The stress-rupture strengths of these alloys are summarized in the following table. Stresses for 1000 hour rupture life are extrapolated values based on tests usually not exceeding 300 hours.

<u>Steel</u>	<u>Temperature</u>	<u>Stress for 100 hour rupture</u>	<u>Stress for 1000 hour rupture</u>
12	1000 F	44,500	33,000
13	1000	42,000	33,000
11	1000	37,500	28,000
10	1000	49,500	43,000
1	1000	34,000	26,500
12	1200	23,000	16,000
13	1200	20,000	14,300
11	1200	19,500	14,500
10	1200	21,500	16,300
1	1200	15,400	10,300

(3) Eichelman, G. H. and Hull, F. C.: The Effect of Composition on the Temperature of Spontaneous Transformation of Austenite to Martensite in 18-8 Type Stainless Steel. Trans. ASM 45, 1953, p. 77.

<u>Steel</u>	<u>Temperature</u>	<u>Stress for 100 hour rupture</u>	<u>Stress for 1000 hour rupture</u>
12	1300 F	14,700	10,300
13	1300	13,600	9,500
11	1300	12,100	8,000
10	1300	14,000	10,200
1	1300	10,800	7,000

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III. Effect of Structure and Composition on the Strength Properties of Stainless Steel

Eight twenty-five pound ingots have been prepared by melting Armo iron, electrolytic nickel, and ferro-chromium in an induction furnace, de-oxidizing with Mn and Si and casting into graphite molds. The Ni content of these ingots was maintained fairly constant at 8 percent while the chromium and the carbon contents were varied as can be seen in the following table:

<u>Heat No.</u>	<u>% Chromium</u>	<u>% Nickel</u>	<u>% Carbon</u>	<u>% Nitrogen</u>
3	17.30	8.28	0.020	0.020
15	16.49	8.12	0.067	0.030
9	17.24	8.18	0.127	0.030
21	18.52	8.04	0.080	0.030
20	18.51	8.13	0.100	0.030
4	19.60	8.46	0.029	0.028
19	19.75	8.12	0.060	0.030
7	19.60	8.05	0.113	0.031

The ingots were hot forged to 1/2 inch diameter rod which in turn was solution treated at 2000 degrees F for 1/2 hour and then quenched in water. Metallographic samples of each heat were examined. The grain size was found to be fairly constant for all the different heats. The steels were found to be chiefly austenitic but a few had a small amount of delta ferrite and martensite. Thirty standard 0.250 inch diameter by 1 inch gage tensile specimens were machined from each heat of steel and stress-rupture tests were conducted at three temperatures, 1000, 1200, and 1300 degrees F. Rupture lives were determined from six minutes to 100 hours at the lower two temperatures and from six minutes to 500 hours at 1300 degrees F. The 500 hour tests are still in progress. The rupture life data obtained so far are summarized in the following table.

Heat No.	<u>1000 F</u>		<u>1200 F</u>		<u>1300 F</u>	
	<u>10 hr.</u>	<u>100 hr.</u>	<u>10 hr.</u>	<u>100 hr.</u>	<u>10 hr.</u>	<u>100 hr.</u>
3	44,000 psi	39,000 psi	26,000 psi	19,000 psi	18,000 psi	11,000 psi
15	55,000	44,000	27,000	20,000	19,000	13,000
9	58,000	48,000	29,500	21,000	19,000	14,000
21	57,000	42,500	26,000	19,000	19,000	12,000
20	55,000	45,000	28,000	20,000	18,000	13,000
4	46,500	40,000	27,000	20,500	19,000	13,000
19	55,000	43,000	29,500	20,500	21,000	14,500

After testing, the test bars were sectioned and micrographic studies are being conducted to determine structural changes such as: recrystallization, grain growth, sigma or ferrite formation, and carbide precipitation. Allowing for these structural and chemical changes, it will be possible to determine the effect of chromium and carbon plus nitrogen content on the high temperature properties.

It is possible to vary the structure of several of these stainless steels by cooling below their M_s and thereby introducing martensite into the structure. The M_s values for these steels are now being determined by measuring the electrical resistance of the steel as a function of the temperature by means of a Kelvin Double Bridge. In the following table are presented the M_s data determined up to the present time, and the M_s temperature as calculated from a formula based on the chemical composition which was determined by Reichman and Hull⁽³⁾.

<u>Heat No.</u>	<u>M_s, calculated</u>	<u>M_s, experimental</u>
3	-20 F	-40 F
15	-60	not accurately determined
9	-315	not detected down to -320 F
21	-240	" " " "
20	-300	" " " "
4	-225	" " " "
19	-280	" " " "
7	-430	" " " "

The tempering of martensite can be followed by resistance measurements and the structures present at the testing temperatures determined by means of additional X-ray and metallographic studies. In this connection a high temperature

X-ray camera has been used to follow the disappearance of the martensite line.

The heating and cooling rate for the resistivity tests was chosen at 35 degrees F/min since it was found that faster rates did not allow sufficient time for accurate measurements. At this rate the complete reversion of martensite to austenite has been observed to occur in the temperature range of 1200 to 1400 degrees F for all the steels tested. Isothermal resistivity tests have been conducted for one steel from 1000 to 1400 degrees F and from the reaction rates the activation energy was determined to be approximately 10,000 cal. Further experimentation is now in progress to determine activation energies for different steels and the amounts of martensite present at various temperatures for different steels. Stress rupture tests then will be run with specimens cooled below their M_s so as to ascertain the effect of an acicular martensite phase on the high temperature strength properties. In particular, heats numbers 3 and 15 look promising for such experimentation.